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TECHNICAL NOTE

EXPLORATORY INVESTIGATION OF SEVERAL COATED AND UNCOATED

METAL, REFRACTORY, AND GRAPHITE MODELS IN A

3,800° F STAGNATION TEMPERATURE AIR JET

By Otto F. Trout, Jr.

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

TECHNICAL NOTE D-190

EXPLORATORY INVESTIGATION OF SEVERAL COATED AND UNCOATED

METAL, REFRACTORY, AND GRAPHITE MODELS IN A 3.800° F STAGNATION TEMPERATURE AIR JET

By Otto F. Trout, Jr.

SUMMARY

An exploratory investigation was conducted on several coated and uncoated metal, graphite, and refractory materials models to determine their reaction in a high-velocity hot airstream having stagnation temperature of approximately 3,800° F for periods of time up to 60 seconds. Tests on the molybdenum models indicate that unprotected molybdenum oxidizes rapidly, whereas the metal protected by various coatings successfully resisted rapid oxidation for short periods of time. Some other coatings on molybdenum were not successful. Tests on various grades of graphite produced rapid oxidation when the material was heated to a high temperature. Siliconized graphite and silicon carbide bonded graphite exhibited better oxidation resistance than plain graphite. Refractory models of silicon carbide, zirconium boride, and siliconized boron showed little change during the tests. A description of the models and the results of the tests performed on them are presented in the present paper.

INTRODUCTION

The search for materials to withstand the heating of increased speeds of flight in the atmosphere has resulted in the demand for increased research on existing materials. Even in the range of 2,000° to 3,000° F little is known of the reaction of materials due to aerodynamic heating. There is a demand for super materials to withstand temperatures of several thousand degrees Fahrenheit in the atmosphere; however, at the present time no scientific breakthrough has occurred in the study of basic properties of matter or solid-state physics for developing such super materials. For the present, technological advances in materials will consist of improvements of existing known substances and combinations thereof.

High melting point metals such as tungsten, tantalum, niobium, and molybdenum have the disadvantage that they oxidize rapidly in air at temperatures below their melting points (refs. 1, 2, and 3). In general, these oxides are volatile or melt at temperatures below the melting point of the basic metal and are generally porous, offering little protection from oxidation. The usefulness of these metals for high-temperature applications in air depends upon the development of coatings or platings which will protect them from oxidation and ignition.

In this series of tests, molybdenum was tested in a hot air jet with platings of chromium and nickel and coatings of molybdenum disilicide, aluminum oxide, zirconium oxide, and nickel aluminide. A model of tungsten was tested with a nickel chromium plating.

Since more information is needed about the lesser known ceramics at high temperatures in air, models of zirconium boride, titanium boride, and siliconized boron were tested. Silicon carbide, the properties of which are well known, was also tested. Carbon is of interest because of its high sublimation temperature and its high heat of sublimation. Because carbon oxidizes rapidly at higher temperatures in air, several grades of graphite were tested to determine relative oxidation resistance. Protective coatings of silicon dioxide, silicon carbide bonded graphite, zirconia, alumina, chrome plate, and platinum plate on graphite were also tested to determine their ability to protect carbon from oxidation.

Very little information is presently available on the reaction of materials at higher temperatures under dynamic conditions in air. The purpose of present tests was to make a qualitative determination of the ability of several materials to withstand heating in a high-temperature, high-velocity airstream. Each model was tested for approximately 60 seconds or until it was damaged in a Mach number 2.0 airstream having a stagnation temperature of approximately 3,800° F and stagnation pressure of 105 psia.

The results of the tests of several coated and uncoated molybdenum, carbon, and ceramic models are presented in the present paper.

TESTS AND METHODS

Figure 1 presents diagrams of model configurations 1 and 2. All the models used in this series of tests were configuration 1 except the models of zirconium boride and siliconized boron which are represented by configuration 2. Figure 2 presents a photograph of the assembly of configuration 1 on the adapter and sting for mounting the model in the jet. Each model was tested in the laboratory-scale ceramic-heated air jet (fig. 3) in a Mach number 2.0 air jet at an average stagnation

temperature of about 3,800° F and 105 psia stagnation pressure for approximately 60 seconds or until the model failed. A more detailed description of the test facility is provided in references 4 and 5.

Photographs of each of the models were taken before and after each test. High-speed motion pictures of each test were made to observe the reaction of the models in the hot airstream.

A description of the materials from which each of the models was constructed is provided in table I. All of the molybdenum models were constructed of a molybdenum alloy containing 0.5 percent titanium.

RESULTS AND DISCUSSION

The results of the present tests are tabulated in table I and photographs of the model and tests are presented in figures 4 to 28.

From the present series of tests it is evident that it is possible to protect molybdenum from oxidation for short periods of time by various coatings - such as, chrome plate, Chromalloy W-2, nickel aluminide, and flame-sprayed molybdenum disilicide. Small imperfections in the coating did not appear to cause rapid failure as evidenced by the molybdenum model with a small hole drilled through the coating for one of the tests. Further investigation of those coatings which failed during the tests, such as chrome nickel plate, flame-sprayed zirconia molybdenum laminate and flame-sprayed alumina molybdenum laminate, and unprotected molybdenum, was not made. Some of the factors which may cause failure of a coating can be difference in thermal expansion between the coating and base material, gaseous diffusion through the coating, the reaction between the coating and base material, and spalling of the coating.

Of the ceramic models tested, only the titanium boride model failed under test conditions, and this is not conclusive since the model was slightly cracked before the test. Refractory models of silicon carbide, zirconium boride, and siliconized boron withstood the heating during the tests. Even though these ceramics withstood high temperatures, it would be necessary to test them in larger sections for thermal shock in order to determine their usefulness.

Tests on the graphite models indicated that graphite oxidized rapidly when heated in a hot air jet for the different grades tested. Graphite can be protected from oxidation by various means, some of which were tried in the present series of tests. Silicon carbide bonded graphite appeared to have much better oxidation resistance than ordinary graphite. Siliconized graphite also had good oxidation resistance. Tests on the other coatings on graphite produced failure of the coatings

in some manner, although each showed some improvement over plain graphite. Improvements of coatings could probably be made to protect carbon from oxidation for longer periods of time.

The present series of tests indicate that presently known materials can be used to withstand high-velocity high-temperature air at the conditions of the tests.

CONCLUSIONS

Tests of coated and uncoated molybdenum, tungsten, refractory materials, and graphite models in a Mach number 2.0 air jet having a stagnation temperature of $3,800^{\circ}$ F produced the following results for test durations of approximately 60 seconds:

- 1. Under the test conditions models of molybdenum were successfully protected against oxidation by chrome plate, Chromalloy W-2, nickel aluminide, and flame-sprayed molybdenum disilicide.
- 2. Models of molybdenum protected by chrome nickel plate, flame-sprayed zirconia molybdenum laminate, and flame-sprayed alumina molybdenum laminate, and unprotected molybdenum failed during the test.
- 3. Several grades of graphite were tested, all of which oxidized rapidly when heated in the hot air jet. Siliconized graphite and silicon carbide bonded graphite were superior to graphite in oxidation resistance.
- 4. Refractory models of silicon carbide, zirconium boride, and siliconized boron withstood the heating during the tests.

Langley Research Center,
National Aeronautics and Space Administration,
Langley Field, Va., September 10, 1959.

REFERENCES

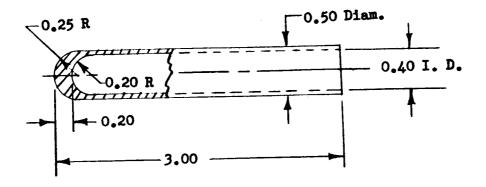
- 1. Hampel, Clifford A., ed.: Rare Metals Handbook. Reinhold Pub. Corp. (New York), 1954.
- 2. Harwood, Julius J., ed.: The Metal Molybdenum. A.S.M. (Cleveland, Ohio), c.1958.
- 3. Grobecker, D. W., ed.: Metals for Supersonic Aircraft and Missiles. A.S.M. (Cleveland, Ohio), c.1958.
- 4. Fields, E. M., Hopko, Russell N., Swain, Robert L., and Trout, Otto F., Jr.: Behavior of Some Materials and Shapes in Supersonic Free Jets at Stagnation Temperatures up to 4,210° F, and a Description of the Jets. NACA RM 157K26, 1958.
- 5. Hopko, Russell N., and Trout Otto F., Jr.: Exploratory Tests of the Behavior of Several Materials in a Supersonic Air Jet at 4,000° F. NACA RM 157E24, 1957.

TABLE I.- DESCRIPTION OF MODELS AND TESTS

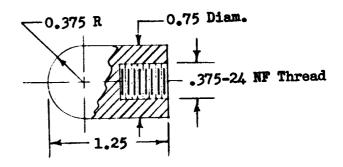
| Description of material | Model configuration | Photographs of model | Figure | Duration of test, sec | Reaction of model during test | Remarks |
|---|------------------------|--------------------------------------|----------------|-----------------------|---|---|
| Molybdenum, unprotected | 1 | Before and after test During test | 4(a) 4(b) | 26 | Oxidation rate increased with surface temperature, ignition occurred at about 26 sec | |
| Molybedenum, electro- plated with 3 mils of nickel undercoat and 2 mils chromium electro- plated on surface | 1 | Before and after test During test | 5(a) 5(b) | 59 | Nickel and chrome plate failed progressively during test | Exposed molybdenum did not ignite during test |
| Molybdenum, coated with Chromalloy W-2 | 1 | Before and after test During test | 6(a) 6(b) | 59 | No failure occurred, surface blackened, slight flow of material on front face | |
| Same as previous test except 1/32-inch hole drilled 1/32 inch deep on center of front face of model | 1 | During test | 6(e) | 60 | Slight oxidation of material in drilled hole, coating intact at end of test | Same appearance as model in previous test |
| Molybdenum, electroplated with 4 mils of chromium | 1 | Before and after test During test | 7(a) 7(b) | 60 | No failure of plating, surface blackened | Only 40 sec of film taken during test |
| Molybdenum, coated with 5 mils of nickel aluminide | 1 | Before and after test During test | 8(a) 8(b) | 58 | No failure occurred, surface was blis- tered and darkened after test | |
| Molybdenum, coated with flame-sprayed molybdenum disilicide | 1 | Before and after test During test | 9(a) 9(b) | 57 | Model remained intact, surface darkened | |
| Molybdenum, coated with alternate layers of flame-sprayed zirconia and molybdenum, 3 layers of zirconia and 3 layers of molybdenum | 1 | Before and after test During test | 10(a) 10(b) | 58 | Coatings failed progressively | Very little mass loss occurred on the exposed molybdenum surface |
| Molybdenum, coated with alternate layers of flame-sprayed alumina and molybdenum, 3 layers of molybdenum and 3 layers of alumina | 1 | Before and after test During test | 11(a) 11(b) | | Coating failed progressively | |
| Tungsten, electroplated with 3 mils of nickel and 2 mils chromium electrodeposited on surface | 1 | Before and after test During test | 12(a) 12(b) | 12 | Failure of plating produced rapid oxidation of tungsten | |
| Phosphate-bonded alumina chromia ceramic rein- forced with spirally wrapped molybdenum wire | 1 | Before and after test During test | 13(a) 13(b) | 60 | Surface spalled causing partial damage | |
| Hot pressed zirconium boride | 2 | Before and after test During test | 14(a) 14(b) | 60 | Model intact after test | |
| Hot pressed siliconized boron machined after pressing | 2 | Before and after test During test | 15(a) 15(b) | 60 | Some spalling of unfinished wall | |
| Hot pressed titanium boride machined after pressing | 1 | Before test During test | 16(a) 16(b) | 59 | Model broke in many pieces at 59 sec | |
| KT silicon carbide | 1 | Before and after test During test | 17(a) 17(b) | 60 | Model remained intact, some exidation of front face | |

TABLE I.- DESCRIPTION OF MODELS AND TESTS - Concluded

| Description of material | Model configuration | Photographs of model | Figure | Duration of test, sec | Reaction of model during test | Remarks |
|--|------------------------|--|----------------------------------|------------------------|--|--|
| Speer grade 8354 graphite | 1 | Before test During test Repeat test | 18(s) 18(b) 18(c) | 23 Repeat 26 | Graphite oxidized rapidly, model broke into many small pieces | |
| Speer grade 3499 graphite | 1 | Before test During test Repeat test with new model | 19(a) 19(b) 19(c) | 21 Repeat 21 | Glowed extremely bright, model broke into many small pieces | |
| Speer grade 8204 graphite | 1 | Before test During test | 20(a) 20(b) | 32 | Glowed extremely bright, model broke into many small pieces | |
| Great Lakes type R graphite | 1 | Before test During test Repeat test with new model | 21(a) 21(b) 21(c) | 22.5 Repeat 27 | Glowed very bright before breaking into many small pieces | |
| ATU graphite | 1 | Before test During test Repeat test | 22(a) 22(b) 22(c) | 16.5 Repeat 16.5 | Glowed very bright before breaking into many small pieces | |
| Siliconized ATJ graphite prepared by vapor depositing silicon on graphite | 1 | Before and after test During test Repeat test with new model | 23(a) 23(c) 23(b) 23(d) | 60 Repeat 60 | Very little change, repeat test showed more damage | Silicon oxide appears to increase oxida- tion resistance of graphite |
| Silicon carbide bonded graphite | 1 | Before test After test During test | 24(a) 24(b) 24(c) | 60 | Some oxide on front surface | |
| ATJ graphite electro- plated with chromium | 1 | Before test | 25(a) 25(b) | 23.5 | Model broke up | |
| ATJ graphite, electro- plated with chromium undercoat, platinum electrodeposited on surface | 1 | Before and after test During test | 26(a) 26(b) | 58 | Model partially failed | Better oxidation resistance than graphite |
| ATJ graphite, coated with alternate layers of flame-sprayed zirconia and molybdenum, 3 coats molybdenum and 2 layers of zirconia | 1 | During test | 27 | 33 | Failure was progressive | |
| ATJ graphite, coated with alternate layers of flame-sprayed alumina and molybdenum, 3 layers of molybdenum and 2 layers of alumina | 1 | Before test During test | 28(a) 28(b) | 32 | Failure was progressive | |



Configuration 1



Configuration 2

Figure 1.- Diagrams of the models. All dimensions are in inches.

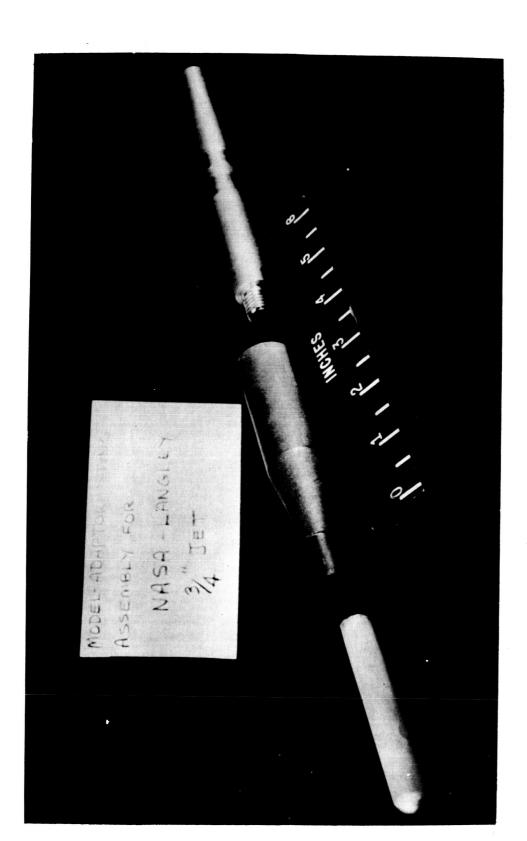


Figure 2.- Photograph of model assembly. L-58-694a

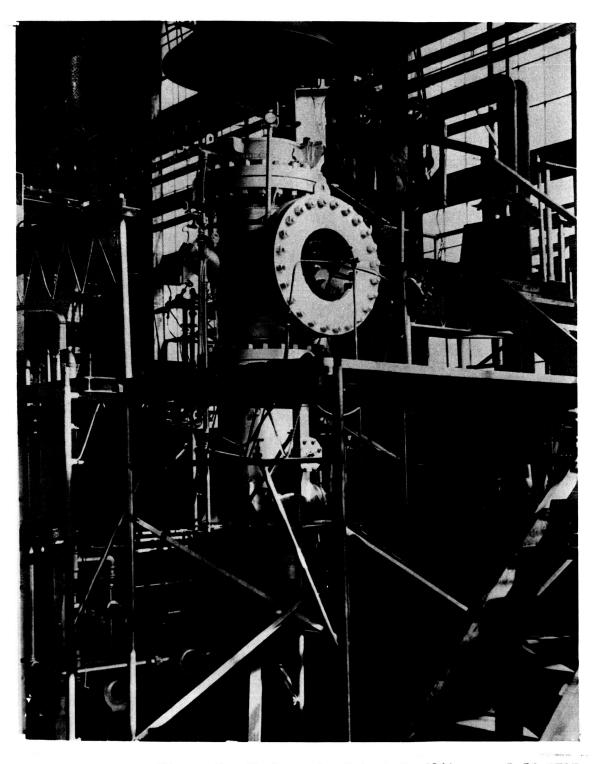
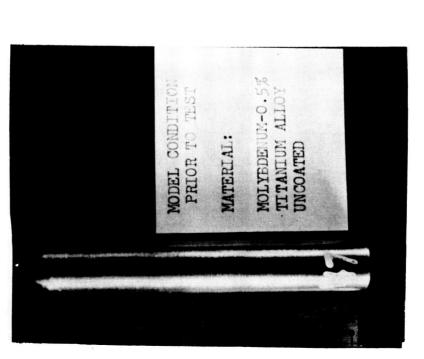


Figure 3.- Photograph of test facility.

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I-59-6400

(a) Before and after the test.

Figure μ .- Unprotected molybdenum model.







7 seconds

O seconds



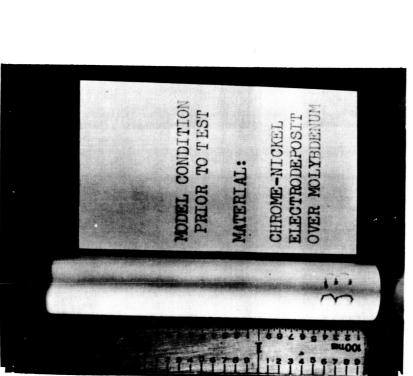


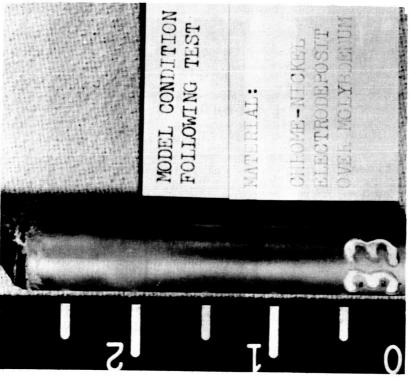
26 seconds

(b) During the test.

L-59-6401

Figure μ .- Concluded.

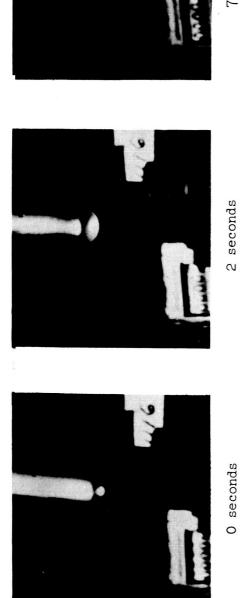


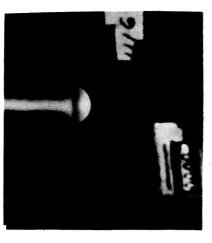


(a) Before and after the test.

I-59-6402

Figure 5.- Nickel chromium electroplated molybdenum model.

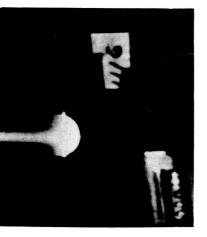




7 seconds



12 seconds



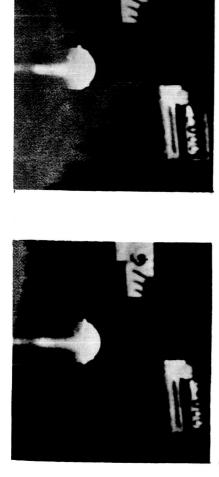
16 seconds



(b) During the test.

Figure 5.- Continued.







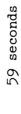


30 seconds

48 seconds





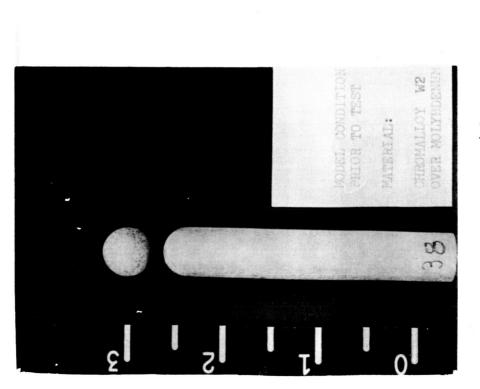


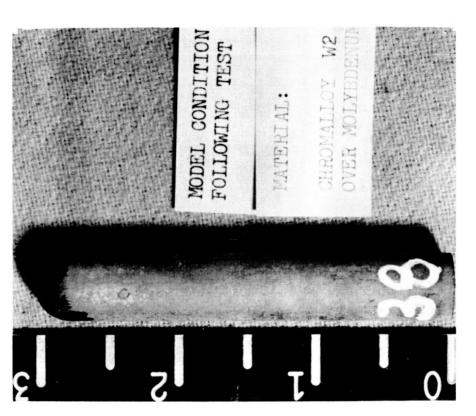


I-59-6404

(b) Concluded.

Figure 5.- Concluded.

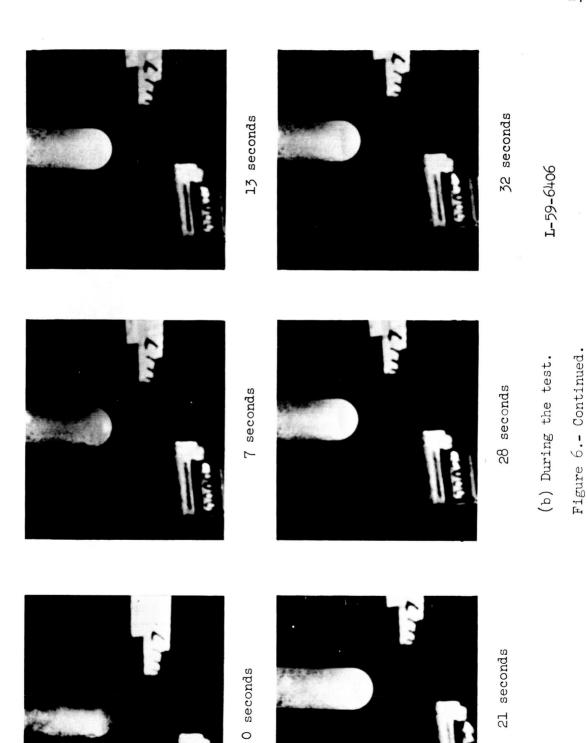




(a) Before and after the test.

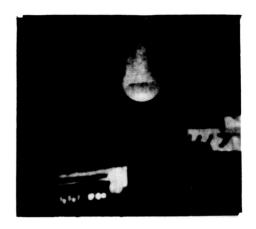
I-59-6405

Figure 6.- Chromalloy W-2 coated molybdenum model.





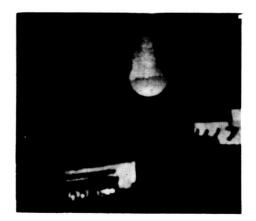
38 seconds



40 seconds



50 seconds

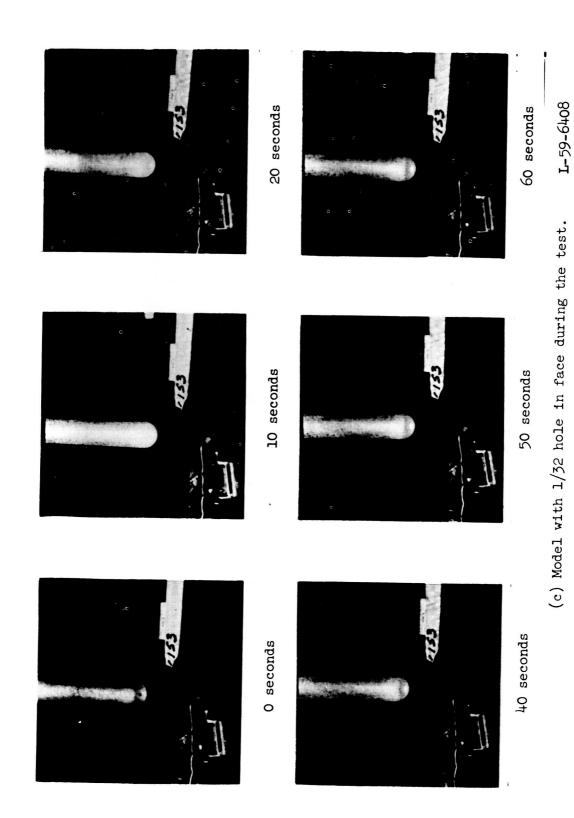


59 seconds

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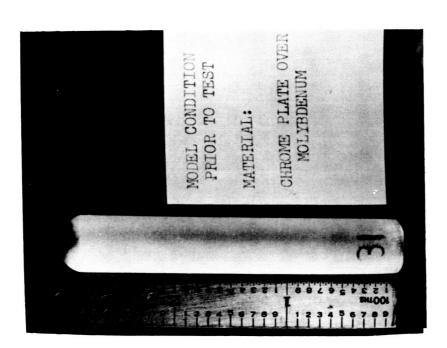
L-59-6407

Figure 6.- Continued.



(2):-1

Figure 6.- Concluded.





(a) Before and after the test.

I-59-6409

Figure 7.- Electrodeposited chromium-plated molybdenum model.

(2)

L-59-6410

Figure 7.- Continued.

(b) During the test.

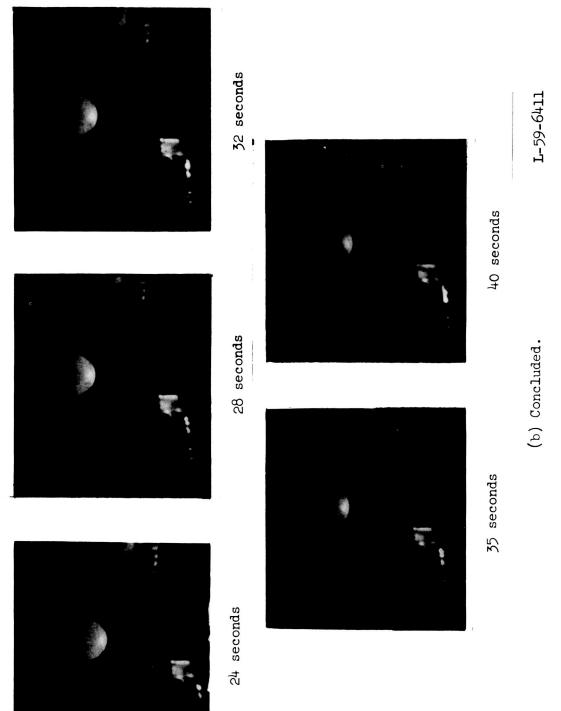
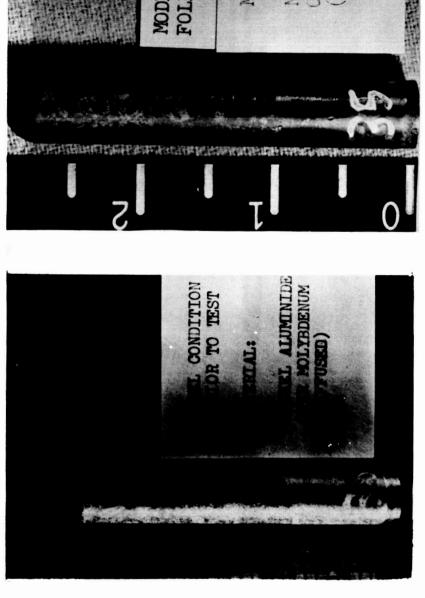
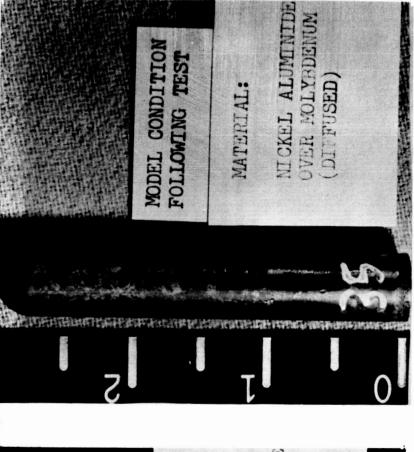


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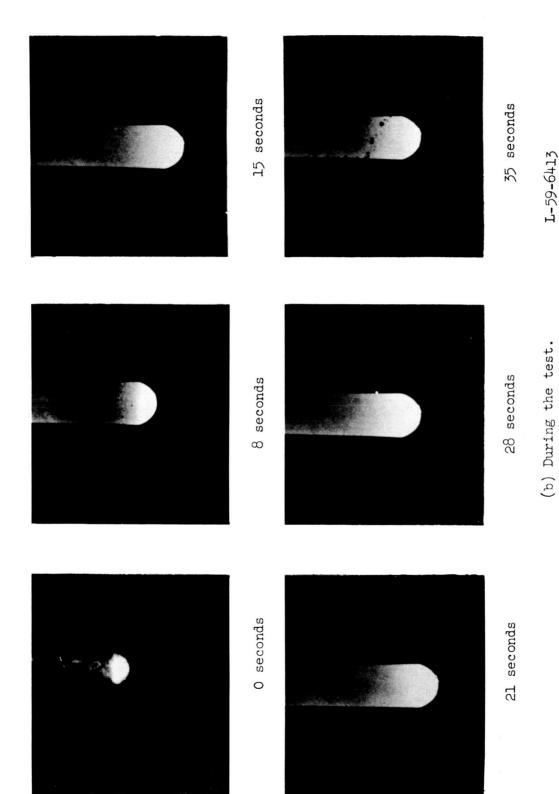




(a) Before and after the test.

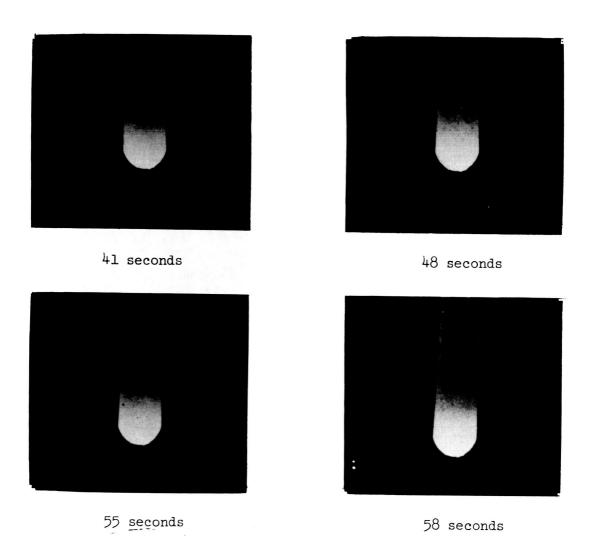
L-59-6412

Figure 8.- Nickel aluminide coated molybdenum model.



L-59-6413

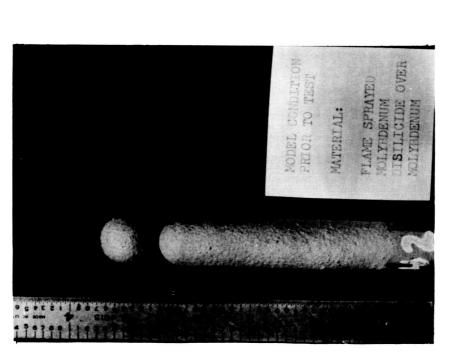
Figure 8.- Continued.



(b) Concluded.

L-59-6414

Figure 8.- Concluded.

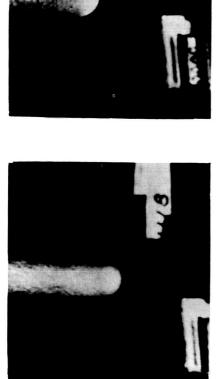




(a) Before and after the test.

L-59-6415

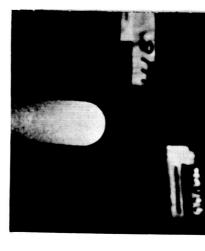
Figure 9.- Flame-sprayed molybdenum disilicide coated molybdenum model.





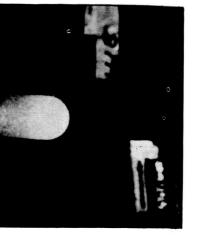
14 seconds

7 seconds



27 seconds





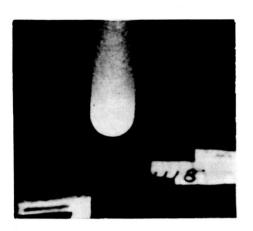
33 seconds

1-59-6416

20 seconds

(b) During the test.

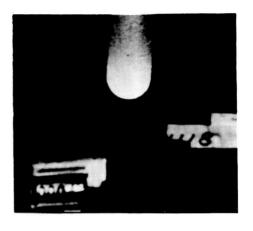
Figure 9.- Continued.



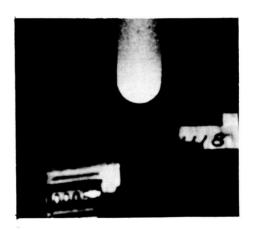
39 seconds



52 seconds



46 seconds

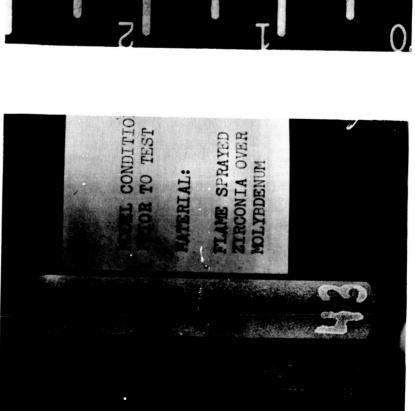


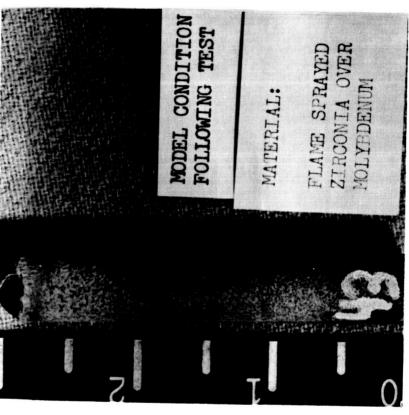
57 seconds

(b) Concluded.

L-59-6417

Figure 9.- Concluded.





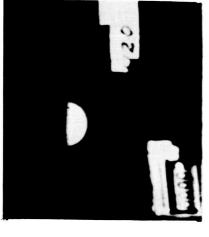
(a) Before and after the test.

L-59-6418

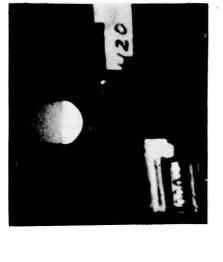
Figure 10.- Flame-sprayed zirconia molybdenum laminate over molybdenum model.



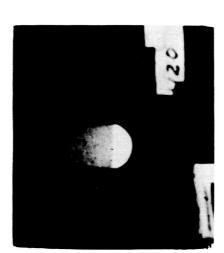
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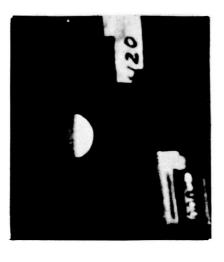
7 seconds



 1^{4} seconds



21 seconds



28 seconds

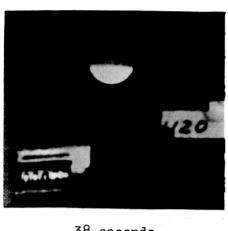
(b) During the test.

Figure 10.- Continued.

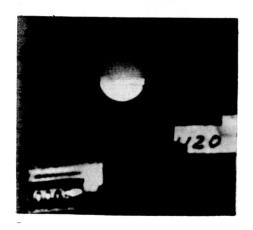


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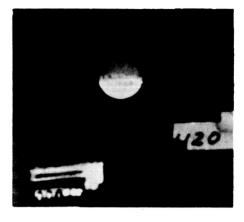
L-59-6419



38 seconds



52 seconds



45 seconds



58 seconds

(b) Concluded.

L-59-6420

Figure 10.- Concluded.

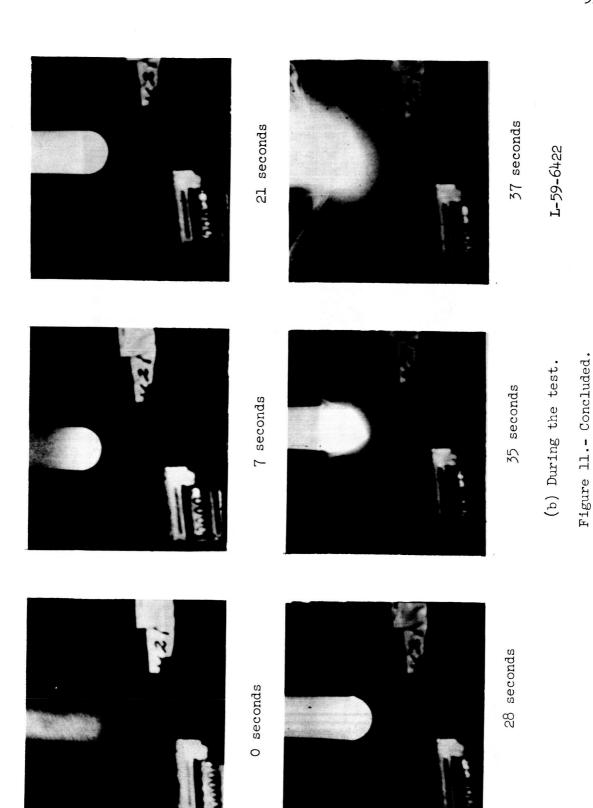


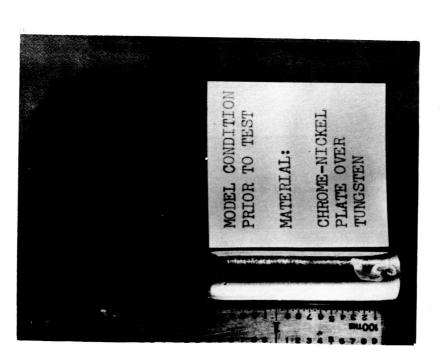


(a) Before and after the test.

L-59-6421

Figure 11.- Flame-sprayed alumina molybdenum laminate coated molybdenum model.







(a) Before and after the test.

I-59-6453

Figure 12.- Chrome nickel plate on tungsten model.



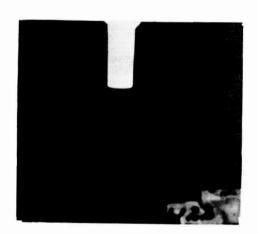
0 seconds



12 seconds



7 seconds



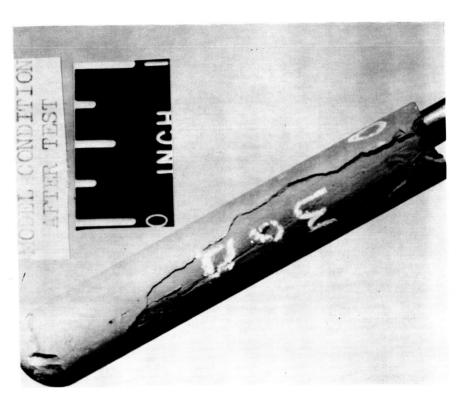
13 seconds

(b) Puring the test.

Figure 12.- Concluded.

L-59-6424



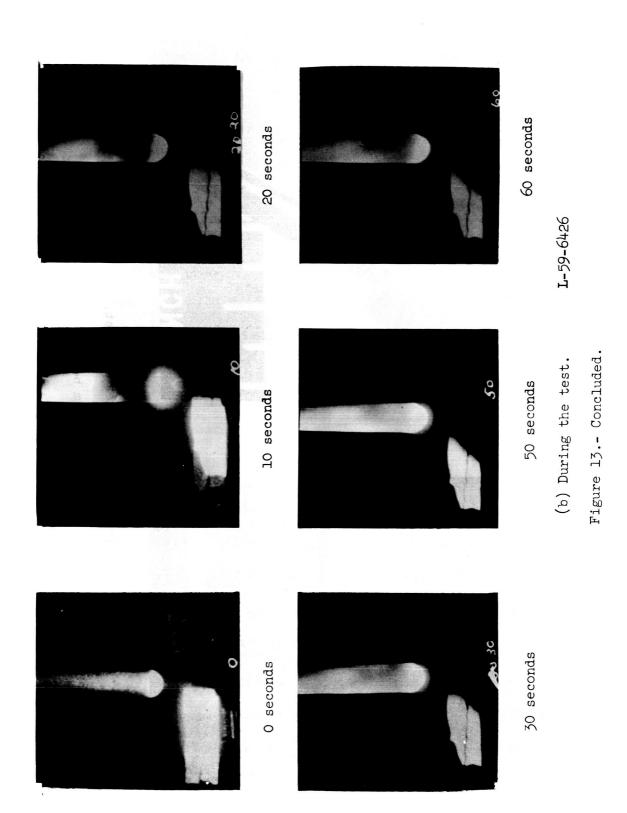


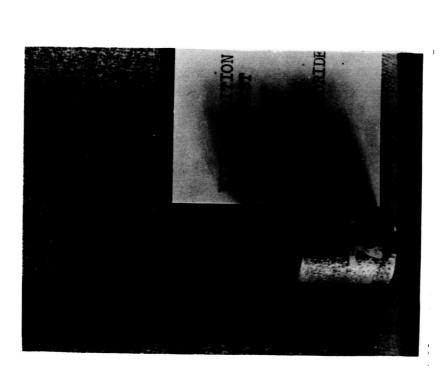
(a) Before and after the test.

1-59-6425

Figure 13.- Phosphate-bonded alumina chromia ceramic model reinforced with spirally wrapped molybdenum wire.

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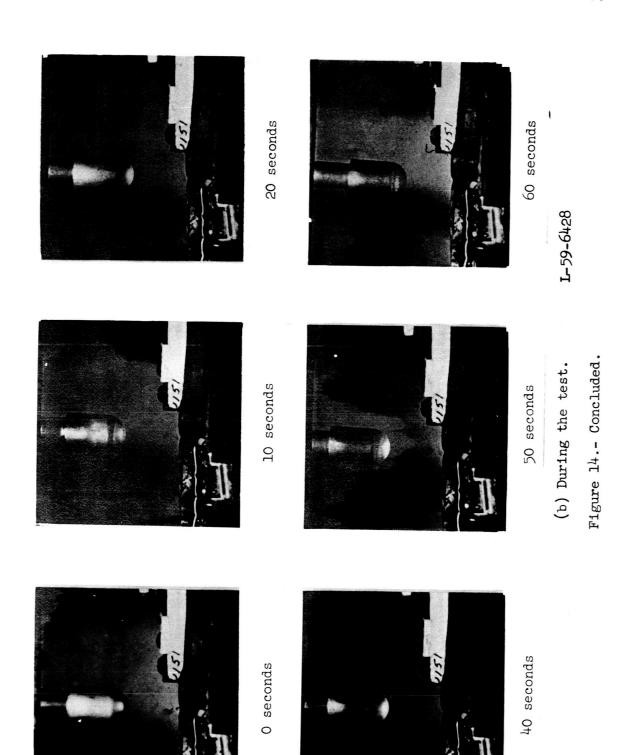


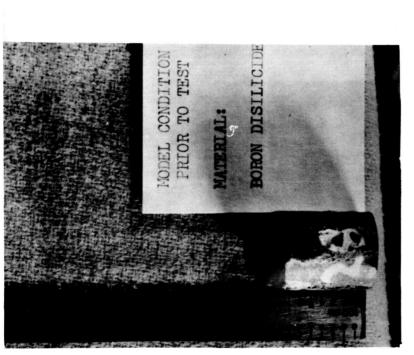


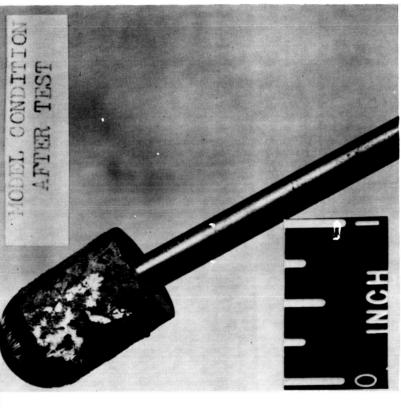
(a) Before and after the test.

L-59-6427

Figure 14.- Zirconium boride model.



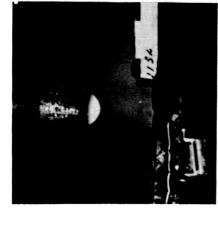




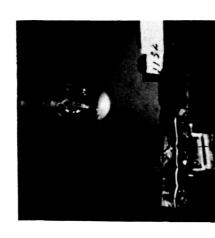
(a) Before and after the test.

I-59-64-29

Figure 15.- Siliconized boron model.



20 seconds



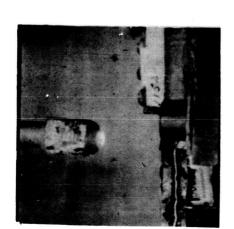
60 seconds



10 seconds



1-59-6430



O seconds

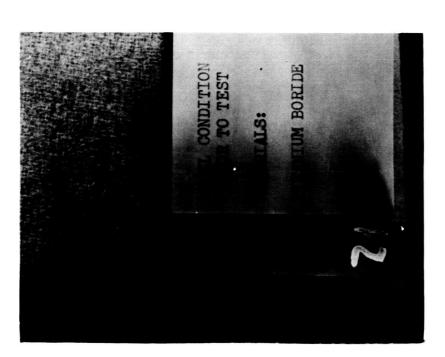


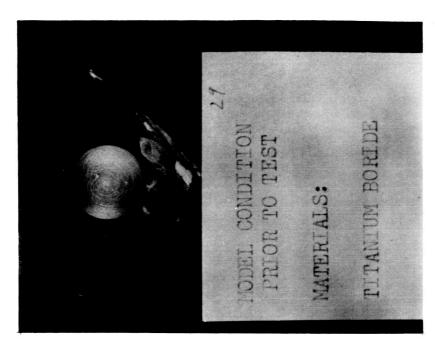
40 seconds

(b) During the test.

50 seconds

Figure 15.- Concluded.



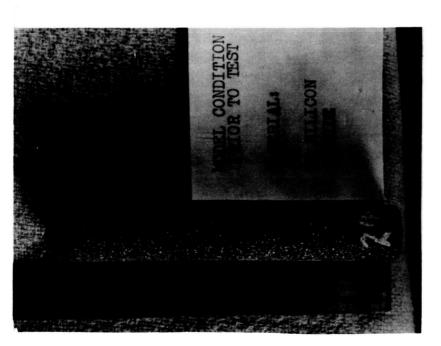


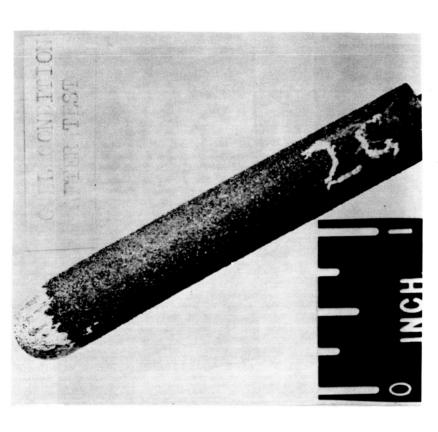
Front view of model

Figure 16.- Titanium boride model.

(2) **-**7

Figure 16.- Concluded.

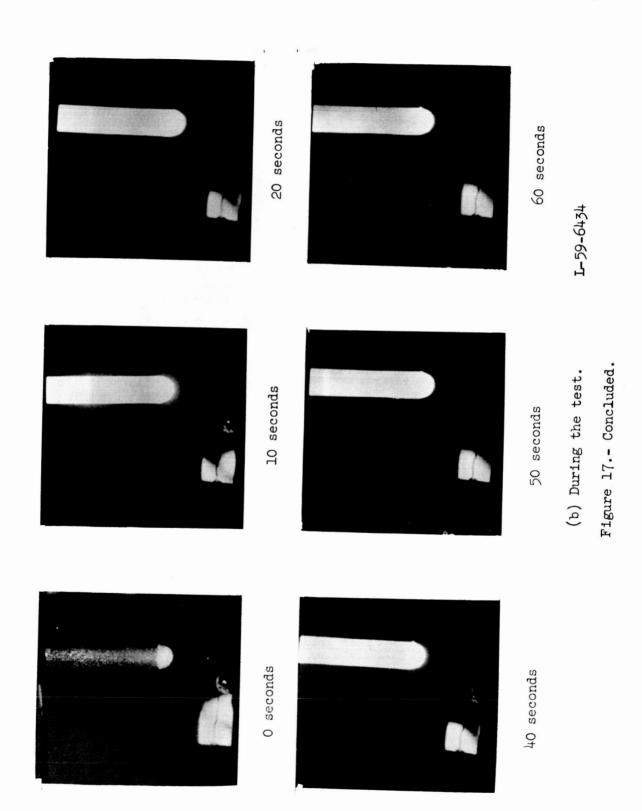




(a) Before and after the test.

1-59-6433

Figure 17.- KT silicon carbide model.



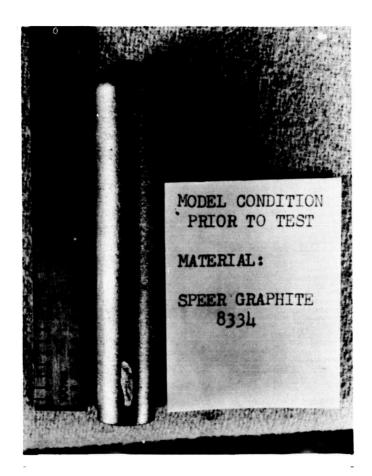
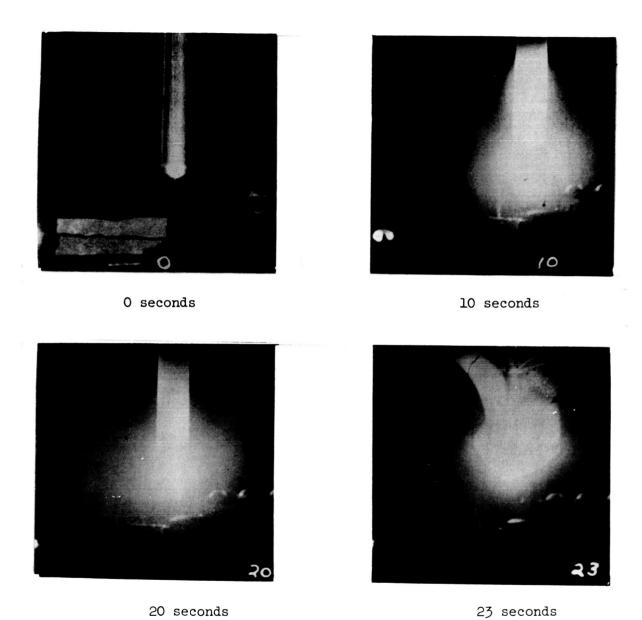


Figure 18.- Speer grade 8334 graphite model.



(b) During the test.

L-59-6436

Figure 18.- Continued.



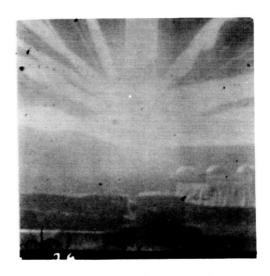
O seconds



20 seconds



10 seconds



26 seconds

(c) During a duplicate test.

Figure 18.- Concluded.

L-59-6437

(a) Before the test.

Figure 19.- Speer grade 3499 graphite model.



0 seconds



20 seconds



10 seconds

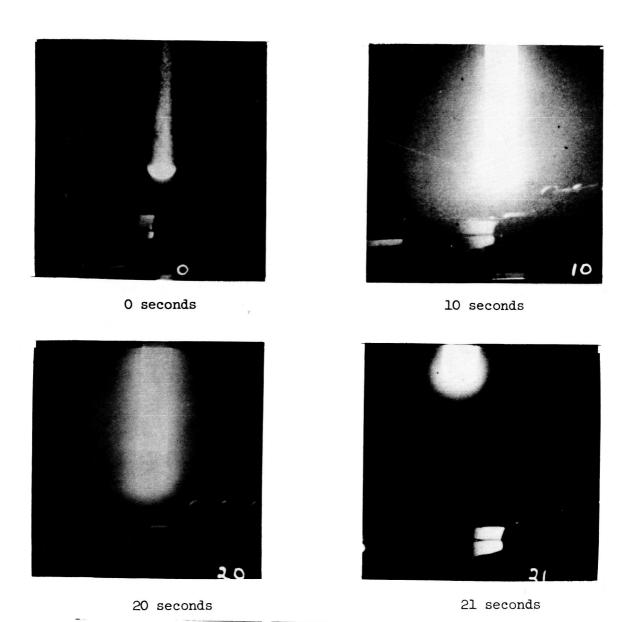


21 seconds

(b) During the test.

Figure 19.- Continued.

L-59-6439



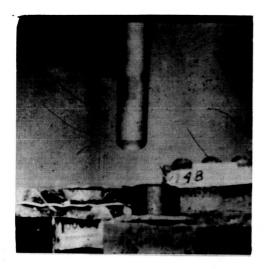
(c) During a duplicate test.

L-59-6440

Figure 19.- Concluded.

(a) Before the test. L-59-6441

Figure 20.- Speer grade 8204 graphite model.



0 seconds



30 seconds



10 seconds



32 seconds

(b) During the test.

Figure 20.- Concluded.

L-59-6442

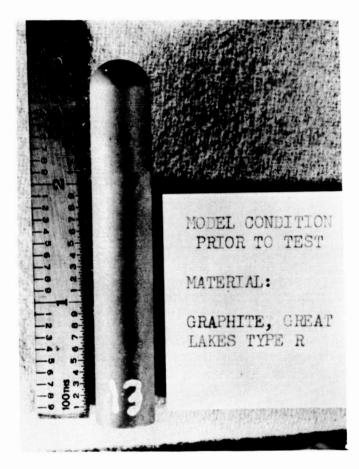


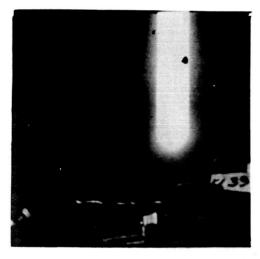
Figure 21.- Great Lakes type R graphite model.

(2)-1

O seconds



22 seconds



10 seconds

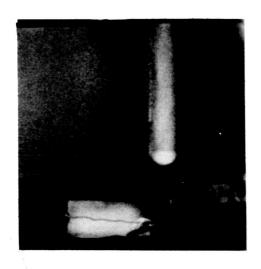


22.5 seconds

(b) During the test.

Figure 21.- Continued.

L-59-6444



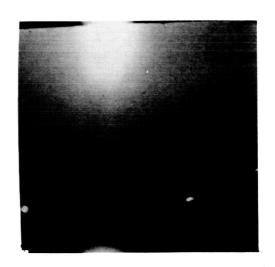
O seconds



20 seconds



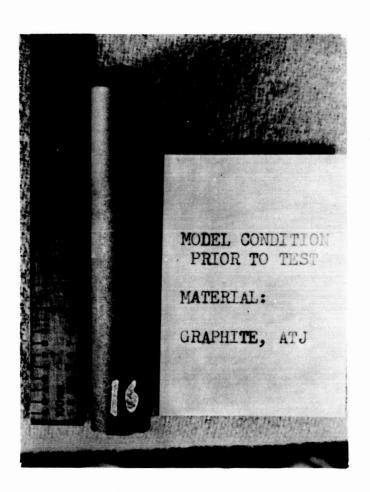
10 seconds



27 seconds

(c) During a duplicate test. L-59-6445

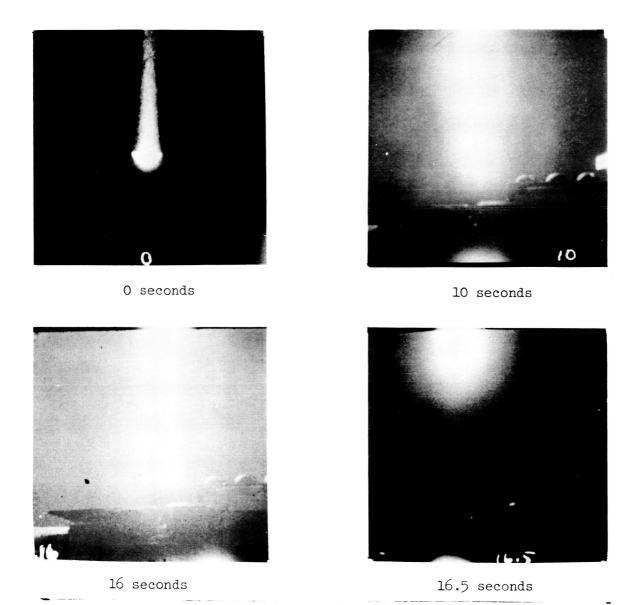
Figure 21.- Concluded.



(a) Before the test.

L-59-6446

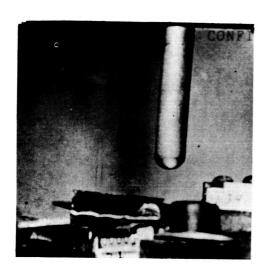
Figure 22.- ATJ graphite model.



(b) During the test.

L-59-6447

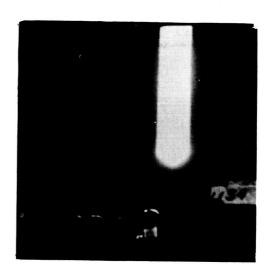
Figure 22.- Continued.



0 seconds



16 seconds



10 seconds

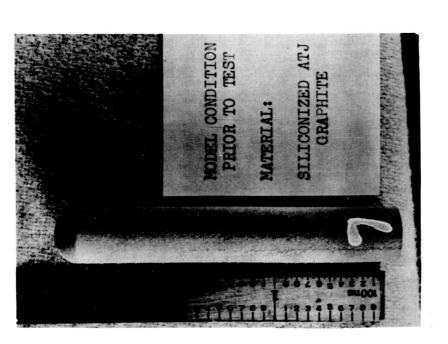


16.5 seconds

(c) During a duplicate test.

Figure 22.- Concluded.

L-59-6448



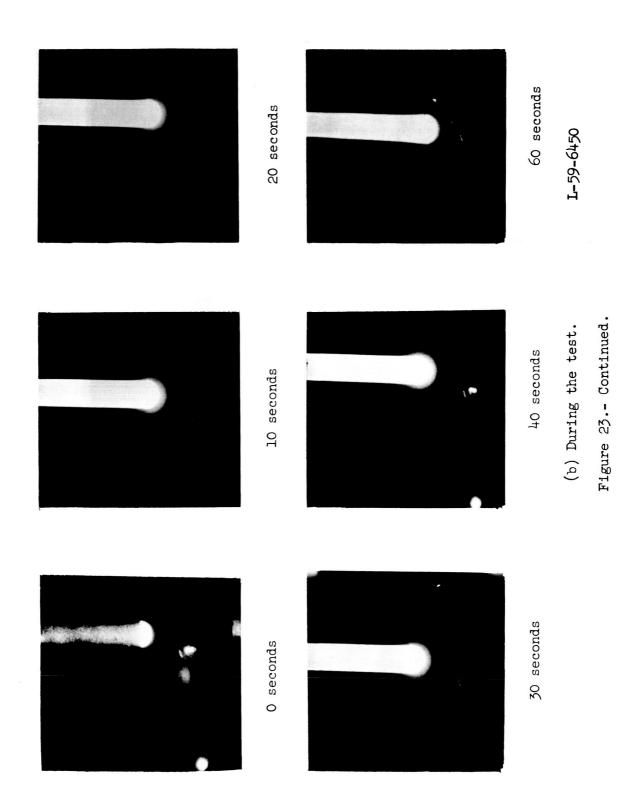


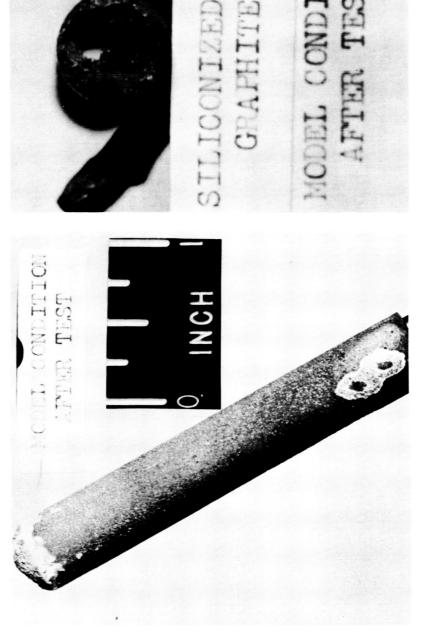
I-59-6

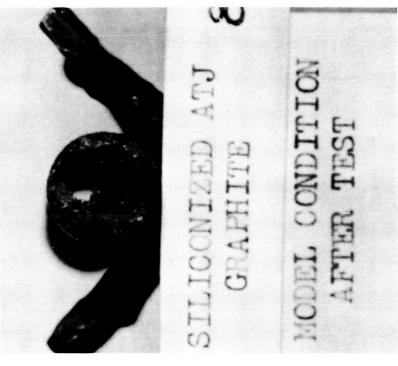
(a) Before and after the test.

I-59-6449

Figure 23.- Siliconized ATJ graphite models.







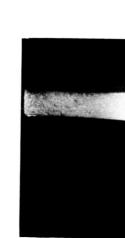
Front view of model

(c) Duplicate model after the test.

L-59-6451

Figure 23.- Continued.

O seconds





L-725

10 seconds



50 seconds



40 seconds



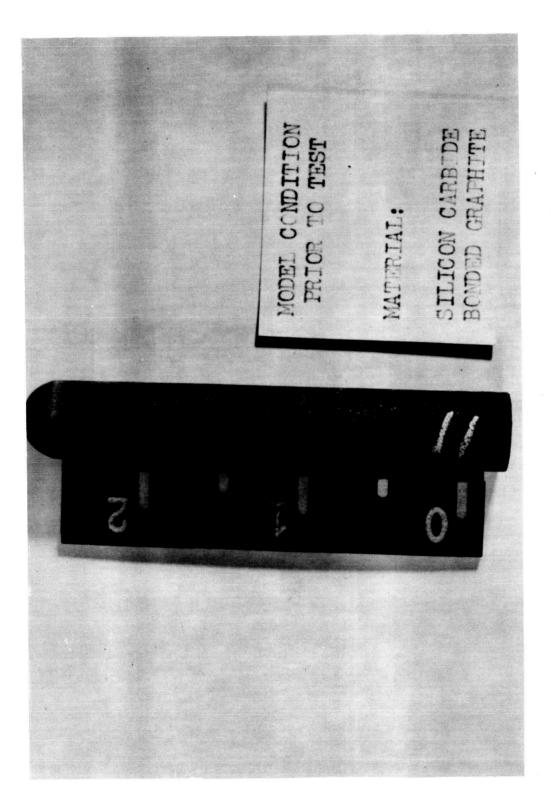


60 seconds

I-59-6452

(d) Duplicate model during the test.

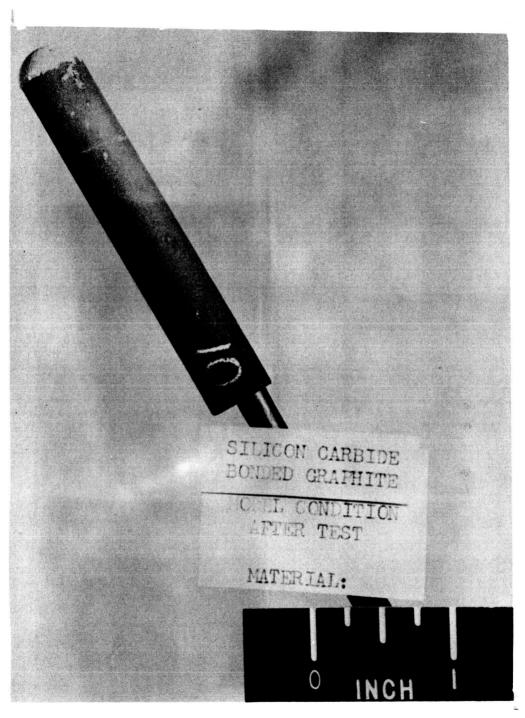
Figure 23.- Concluded.



(a) Before the test.

L-58-1015a

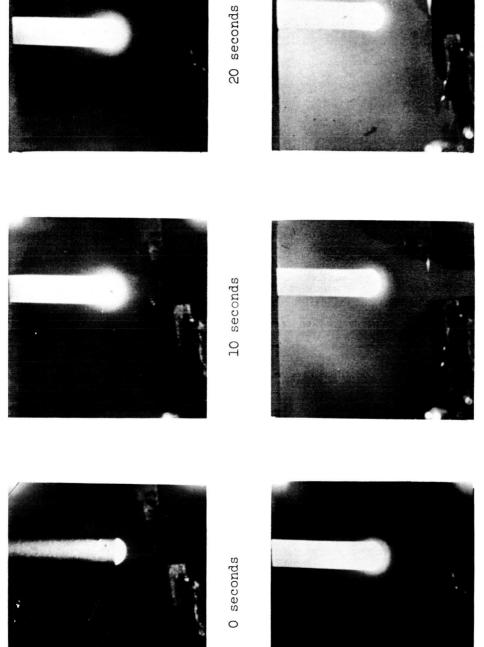
Figure 2μ .- Silicon carbide bonded graphite model.



(b) After the test.

L-59-250

Figure 24.- Continued.







60 seconds

40 seconds

(c) During the test.

50 seconds

Figure 24. - Concluded.

L-59-6453

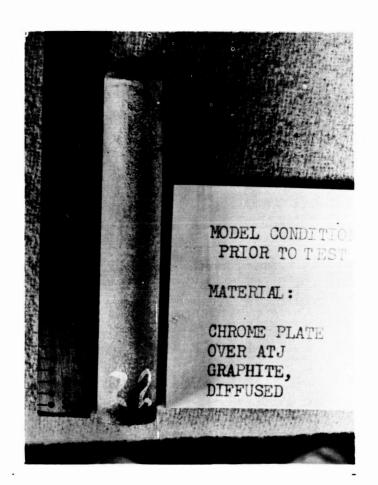


Figure 25.- Chrome plated ATJ graphite model.



0 seconds



20 seconds



10 seconds

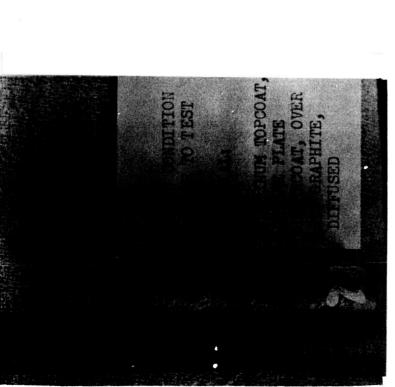


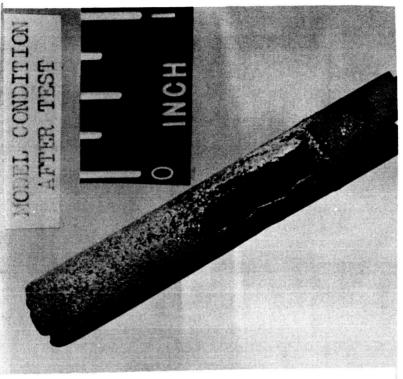
23.5 seconds

(b) During the test.

L-59-6455

Figure 25.- Concluded.



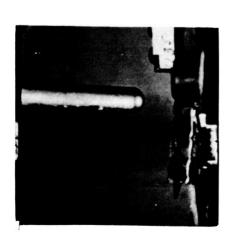


L-725

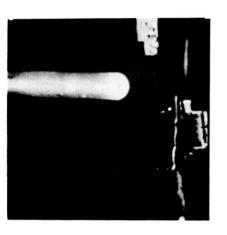
1-59**-**6456

(a) Before and after the test.

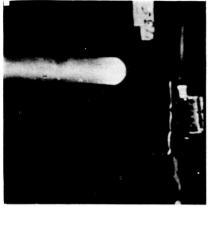
Figure 26.- Platinum chrome plated ATJ graphite model.



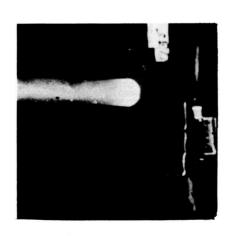
O seconds



10 seconds



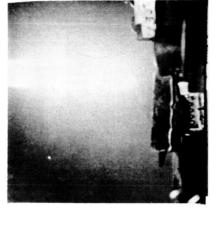
30 seconds



40 seconds



50 seconds



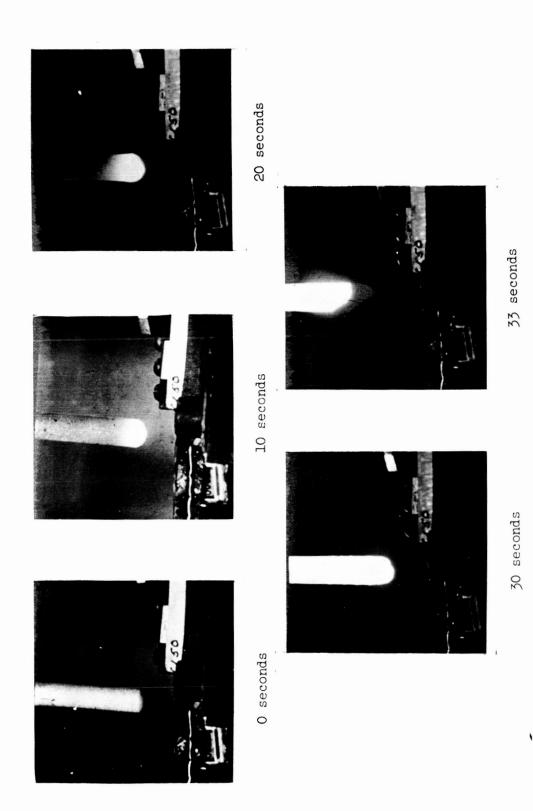
58 seconds

I-59-6457

Figure 26.- Concluded.

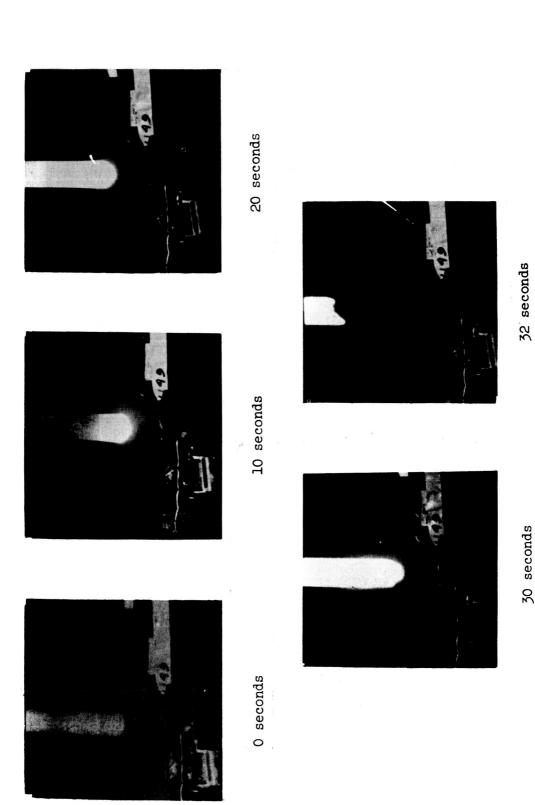
(b) During the test.

F. C



I-59-6458 Figure 27.- Zirconia molybdenum laminate over ATJ graphite model at various times during the test.

Figure 28.- Alumina-molybdenum laminate over ATJ graphite model.



1-59-6460

(b) During the test.

Figure 28.- Concluded.